

# UNIVERSITY OF BIRMINGHAM

## Research at Birmingham

### Data comparing the separation and elution of vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometer (UPSFC-MS/MS) compared to liquid chromatography (LC) and data presenting approaches to UPSFC method optimization

Jenkinson, Carl; Taylor, Angela; Storbeck, Karl-Heinz; Hewison, Martin

DOI:

[10.1016/j.dib.2018.08.027](https://doi.org/10.1016/j.dib.2018.08.027)

License:

Creative Commons: Attribution-NonCommercial-NoDerivs (CC BY-NC-ND)

*Document Version*

Publisher's PDF, also known as Version of record

*Citation for published version (Harvard):*

Jenkinson, C, Taylor, A, Storbeck, K-H & Hewison, M 2018, 'Data comparing the separation and elution of vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometer (UPSFC-MS/MS) compared to liquid chromatography (LC) and data presenting approaches to UPSFC method optimization', *Data in Brief*, vol. 20, pp. 426-435. <https://doi.org/10.1016/j.dib.2018.08.027>

[Link to publication on Research at Birmingham portal](#)

#### General rights

Unless a licence is specified above, all rights (including copyright and moral rights) in this document are retained by the authors and/or the copyright holders. The express permission of the copyright holder must be obtained for any use of this material other than for purposes permitted by law.

- Users may freely distribute the URL that is used to identify this publication.
- Users may download and/or print one copy of the publication from the University of Birmingham research portal for the purpose of private study or non-commercial research.
- User may use extracts from the document in line with the concept of 'fair dealing' under the Copyright, Designs and Patents Act 1988 (?)
- Users may not further distribute the material nor use it for the purposes of commercial gain.

Where a licence is displayed above, please note the terms and conditions of the licence govern your use of this document.

When citing, please reference the published version.

#### Take down policy

While the University of Birmingham exercises care and attention in making items available there are rare occasions when an item has been uploaded in error or has been deemed to be commercially or otherwise sensitive.

If you believe that this is the case for this document, please contact [UBIRA@lists.bham.ac.uk](mailto:UBIRA@lists.bham.ac.uk) providing details and we will remove access to the work immediately and investigate.

Download date: 01. Feb. 2019



ELSEVIER

Contents lists available at ScienceDirect

## Data in Brief

journal homepage: [www.elsevier.com/locate/dib](http://www.elsevier.com/locate/dib)

CrossMark

## Data Article

# Data comparing the separation and elution of vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometer (UPSFC-MS/MS) compared to liquid chromatography (LC) and data presenting approaches to UPSFC method optimization

Carl Jenkinson<sup>a,\*</sup>, Angela E. Taylor<sup>a</sup>, Karl-Heinz Storbeck<sup>b</sup>,  
Martin Hewison<sup>a,c</sup>

<sup>a</sup> Institute of Metabolism and Systems Research, The University of Birmingham, Birmingham B15 2TT, UK

<sup>b</sup> Department of Biochemistry, Stellenbosch University, Stellenbosch 7600, South Africa

<sup>c</sup> CEDAM, Birmingham Health Partners, The University of Birmingham, Birmingham B15 2TT, UK

## ARTICLE INFO

*Article history:*

Received 19 December 2017

Received in revised form

24 July 2018

Accepted 9 August 2018

Available online 15 August 2018

*Keywords:*

Vitamin D

Method development

UPSFC-MS/MS

LC-MS/MS

## ABSTRACT

The data presented is related to the research article “Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS)” (Jenkinson et al., 2018) [1]. This article will include data obtained from method development, optimization and analysis of multiple vitamin D metabolites on an ultra performance supercritical fluid chromatography tandem-mass spectrometry (UPSFC-MS/MS). This includes chromatograms from column screening to confirm the most suitable column for analyte separation. Additionally, further chromatograms and figures compare separation and analyte signal strength during the optimization of other UPSFC parameters. Mass spectra will demonstrate the optimization of MS conditions for the UPSFC-MS/MS method. Chromatogram data from UHPLC vitamin D analysis is also presented in order to compare the separation and elution of vitamin D metabolites using

\* Corresponding author.

E-mail address: [C.Jenkinson@Bham.ac.uk](mailto:C.Jenkinson@Bham.ac.uk) (C. Jenkinson).

UPSFC and UHPLC. This data will highlight the outputs that aid in method development and identifying the separation technique suited for vitamin D quantitation.

© 2018 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## Specifications Table

Subject area	<i>Analytical Chemistry</i>
More specific subject area	<i>Vitamin D and supercritical fluid chromatography</i>
Type of data	<i>Chromatograms Mass spectra Figures</i>
How data was acquired	<i>Method development for the separation and analysis of vitamin D metabolites was performed on Waters ACQUITY UPC<sup>2</sup> and Waters ACQUITY UPLC coupled to a Waters Xevo TQ-MS mass spectrometer.</i>
Data format	<i>Raw and analyzed</i>
Experimental factors	<i>Working standards of vitamin D were prepared in methanol for UPSFC-MS/MS analysis and methanol/water (50/50%) for UPLC-MS/MS analysis. Derivatization of vitamin D metabolites was performed using 4-Phenyl-1,2,4-triazole-3,5-dione (PTAD) and 4-[2-(3,4-Dihydro-6,7-dimethoxy-4-methyl-3-oxo-2-quinoxaliny)ethyl]-3H-1,2,4-triazole-3,5(4H)-dione (DMEQ-TAD).</i>
Experimental features	<i>Comparison of vitamin D metabolite elution and separation between UPSFC and UPLC. Optimization of UPSFC parameters for separation and detection of vitamin D.</i>
Data source location	<i>Birmingham, United Kingdom.</i>
Data accessibility	<i>Data is with this article</i>
Related research article	<i>C. Jenkinson, A Taylor, K. Storbeck, M. Hewison. Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS). Journal of Chromatography B. 2017., 1087–1088 (2018), pp.43–48. doi: 10.1016/j.jchromb.2018.04.025</i>

## Value of the data

- The direct comparison between optimized UPSFC and UPLC methods could provide an insight into which separation technique is best suited for routine analysis of vitamin D and other similar small molecules.
- The data presented from the UPSFC-MS/MS method development and optimization provides a benchmark for future method development approaches using this platform.
- The analytical methods presented incorporate the analysis of multiple active and inactive vitamin D forms across the metabolic pathway. This data will be valuable for clinical assessments in vitamin D health and disease.

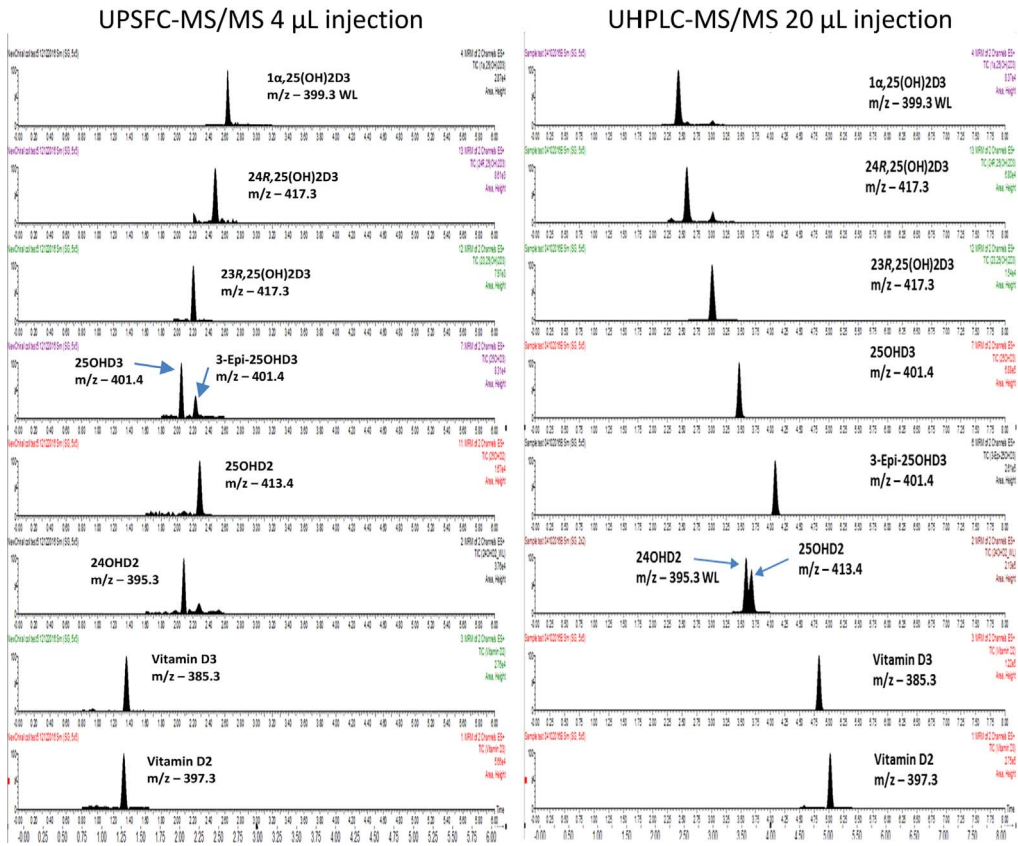


Fig. 1. Chromatogram of vitamin D analytes on UPSFC and UPLC, separated using a Lux Cellulose-2 chiral column.

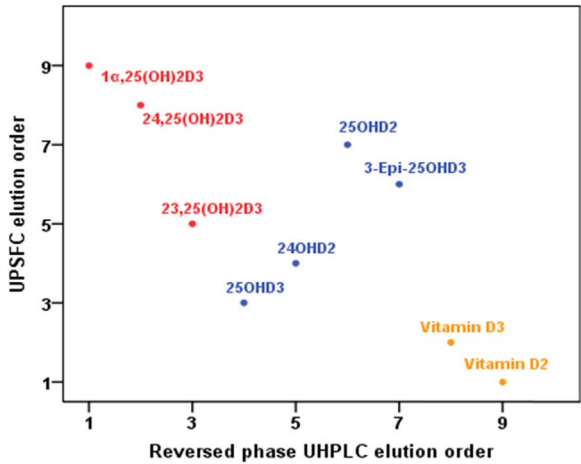


Fig. 2. Comparison in elution order of vitamin D analytes on UPSFC and UPLC.

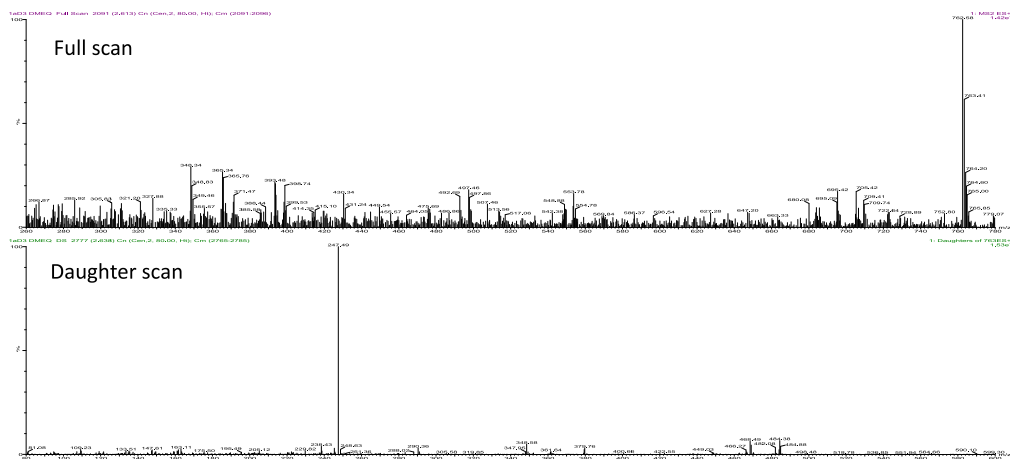


Fig. 3. Mass spectrum of full scan and daughter scans of  $1\alpha,25(\text{OH})_2\text{D}_3$  following derivatization with DMEQ-TAD.

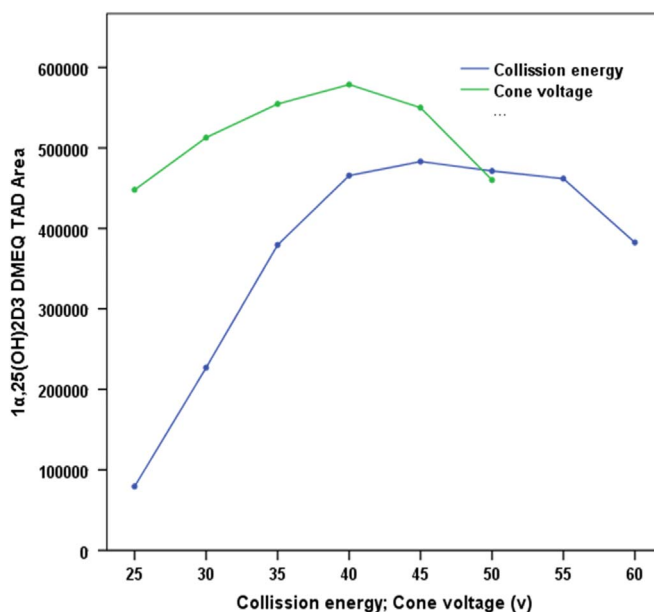


Fig. 4. Signal intensity (area) of  $1\alpha,25(\text{OH})_2\text{D}_3$ -DMEQ TAD at a range of collision energy (25–60) and cone voltage (25–50) values.

## 1. Data

### 1.1. Elution order and chromatography comparison between UPSFC-MS/MS and UPLC-MS/MS

The chromatograms in Section 1.1 compare the chromatography of UPSFC-MS/MS and UPLC-MS/MS for measuring multiple vitamin D metabolites; vitamin D<sub>3</sub>, vitamin D<sub>2</sub>, 25-hydroxyvitamin D<sub>3</sub> (25OHD<sub>3</sub>), 25OHD<sub>2</sub>, 24OHD<sub>2</sub>, 3-epi-25OHD<sub>3</sub>,  $1\alpha,25$ -dihydroxyvitamin D<sub>3</sub> ( $1\alpha,25(\text{OH})_2\text{D}_3$ ), 23,25

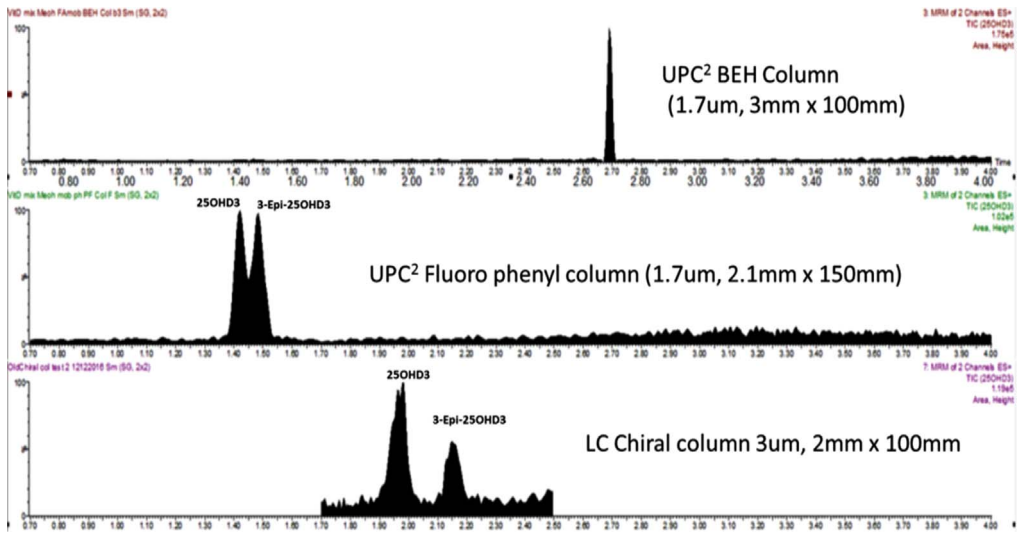


Fig. 5. Column screening on UPSFC to separate 25OHD3 and 3-epi-25OHD3. Both analytes have a mass to charge ( $m/z$ ) of 401.6.

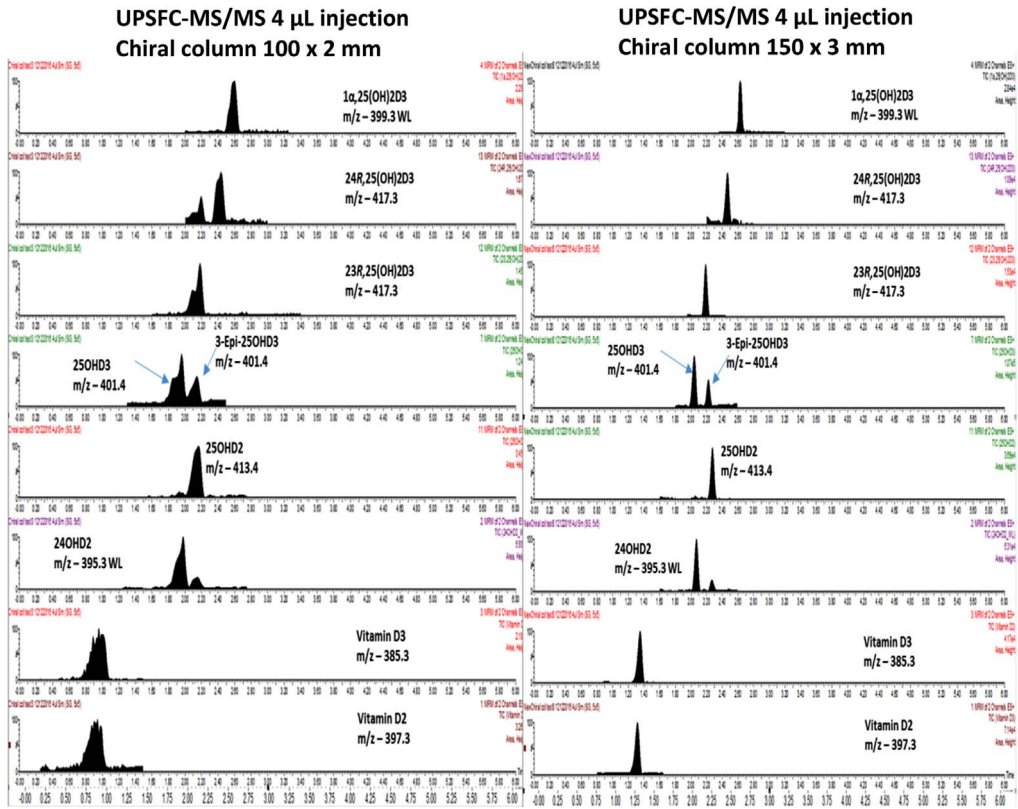
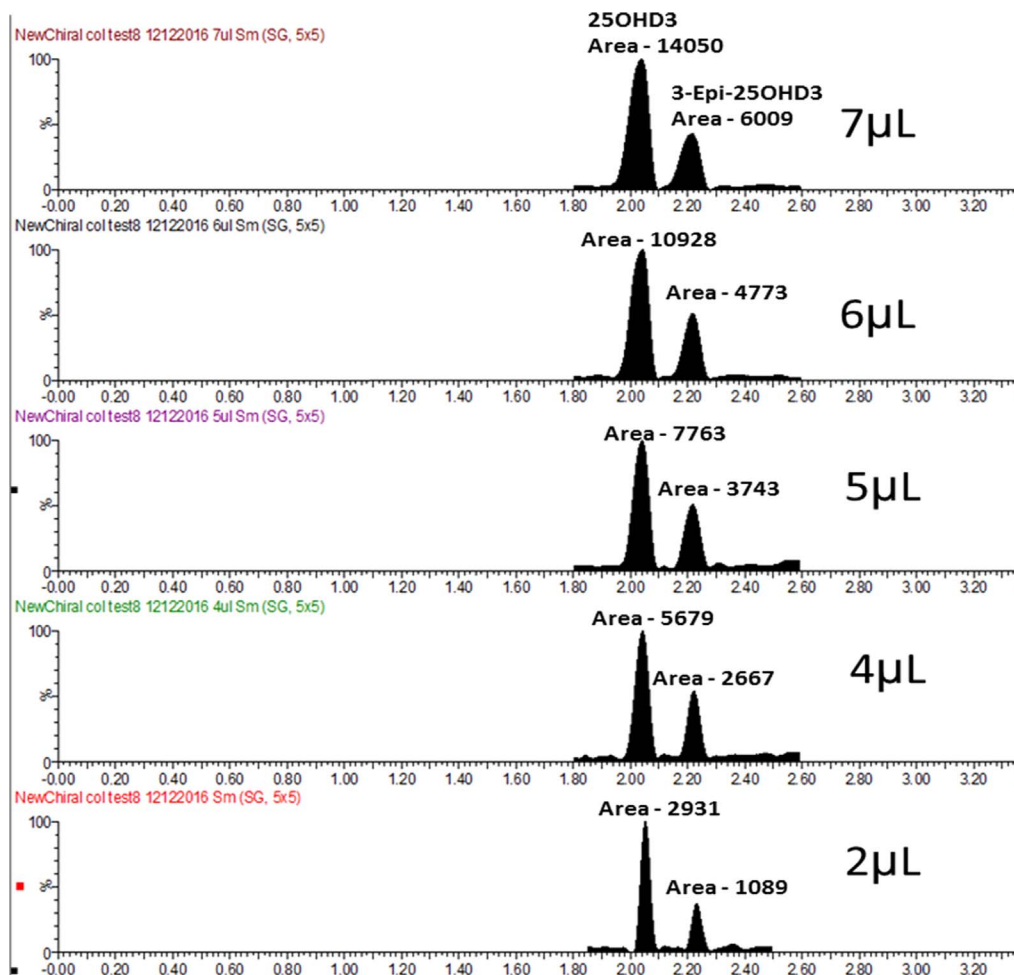


Fig. 6. Chromatogram of vitamin D analytes on Lux Cellulose-2 chiral columns with different dimensions: 100 × 2 mm and 150 × 3 mm.



**Fig. 7.** Separation and signal intensity (area) of 25OHD3 and 3-epi-25OHD3 with increasing sample injection volume (2–7 µL) using a 150 × 3 mm Lux Cellulose-2 chiral column.

(OH)<sub>2</sub>D3 and 24,25(OH)<sub>2</sub>D3 (Fig. 1). The elution order of vitamin D analytes is compared between UPSFC and UPLC in Fig. 2.

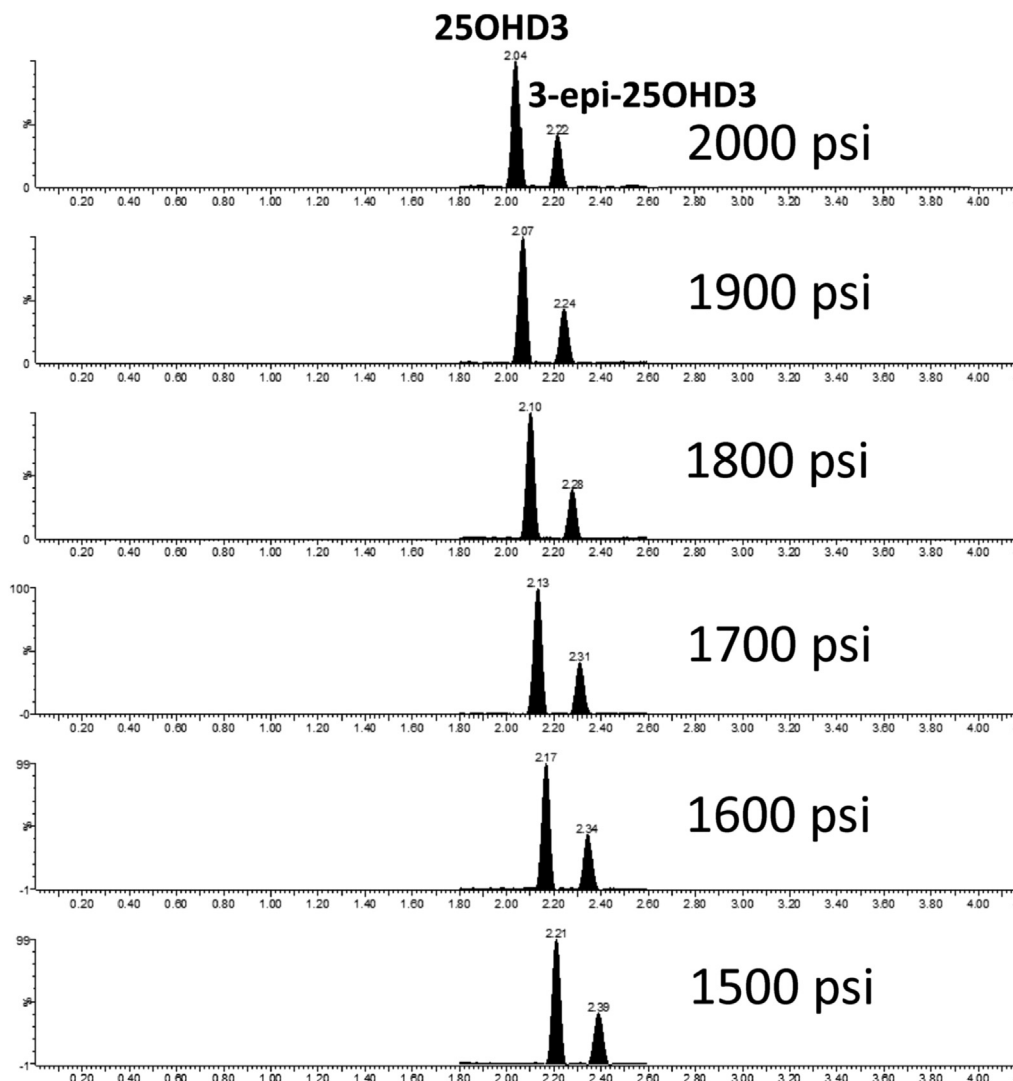
### 1.2. Optimization of mass spectrometry conditions

The data in Section 2 was obtained during the method development and optimization of the UPSFC-MS/MS method. Section 2.1 presents data from the optimization of multiple reaction mode (MRM) parameters using 1 $\alpha$ ,25(OH)<sub>2</sub>D3 derivatized with DMEQ-TAD as an example. The mass spectra from full scan and daughter scan of m/z 762.6 > 247.5 are shown in Fig. 3. The signal intensity of 1 $\alpha$ ,25(OH)<sub>2</sub>D3 DMEQ-TAD is compared with a range of cone voltage and collision energies in Fig. 4 to determine the optimal values.

### 1.3. Optimization of UPSFC column conditions

The chromatograms and figures in section 2.2 relate to the optimization of UPSFC column screening and selection for optimized separation of vitamin D metabolites. The chromatograms





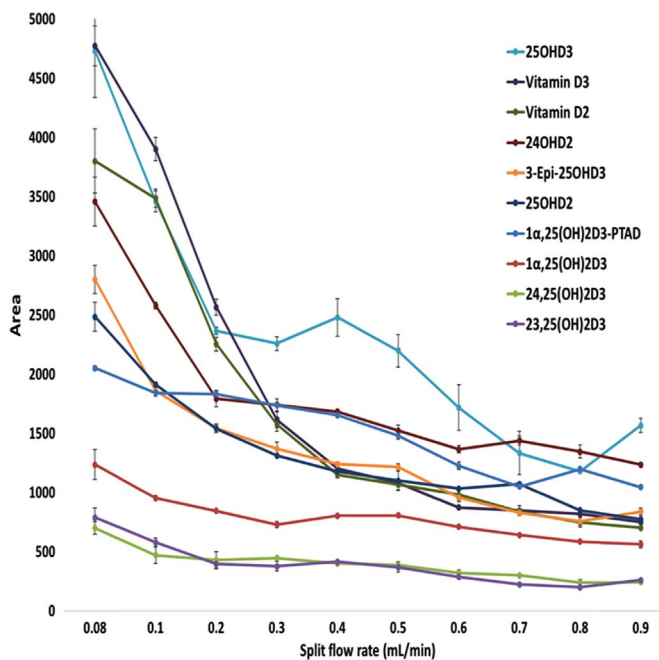
**Fig. 8.** Elution of 25OHD3 and 3-epi-25OHD3 with increasing ABPR (1500–2000 psi) using a 150 × 3 mm Lux Cellulose-2 chiral column.

displayed in Figs. 5 and 6 compare different column chemistries and column size to optimize vitamin D separation by UPSFC.

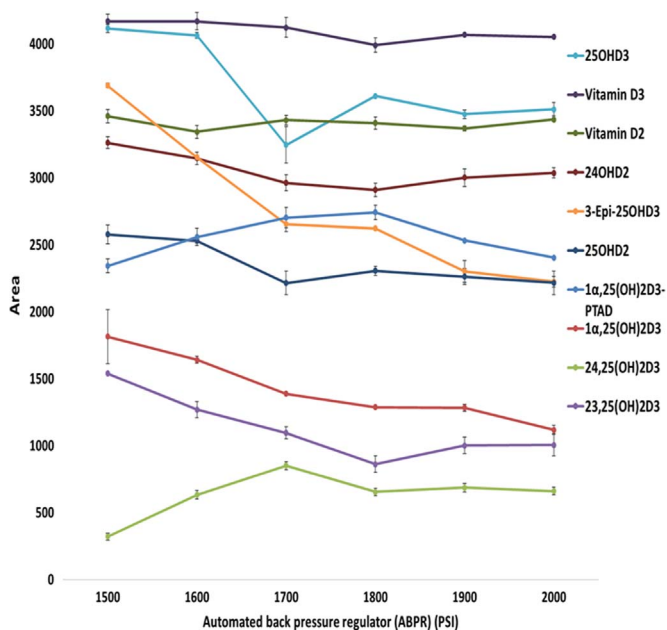
#### 1.4. Optimization of UPSFC method parameters

The chromatographs and figures in section 2.3 are outputs from the optimization of UPSFC parameters for vitamin D analysis. The chromatograms compare the separation and signal intensity of 25OHD3 and 3-epi-25OHD3 with increasing injection volume and atmospheric back pressure regulator (ABPR) in Figs. 7 and 8, respectively. The signal intensity of vitamin D analytes is compared for the optimization of the inlet flow rate, ABPR, column temperature and solvent for sample reconstitution in Figs. 9–11 respectively (Fig. 12).





**Fig. 9.** Analyte areas of vitamin D metabolites with increasing split flow rate (0.08–0.9 mL/min) containing methanol 0.1% formic acid.



**Fig. 10.** Analyte areas of vitamin D metabolites with increasing ABPR (1500–200 psi).

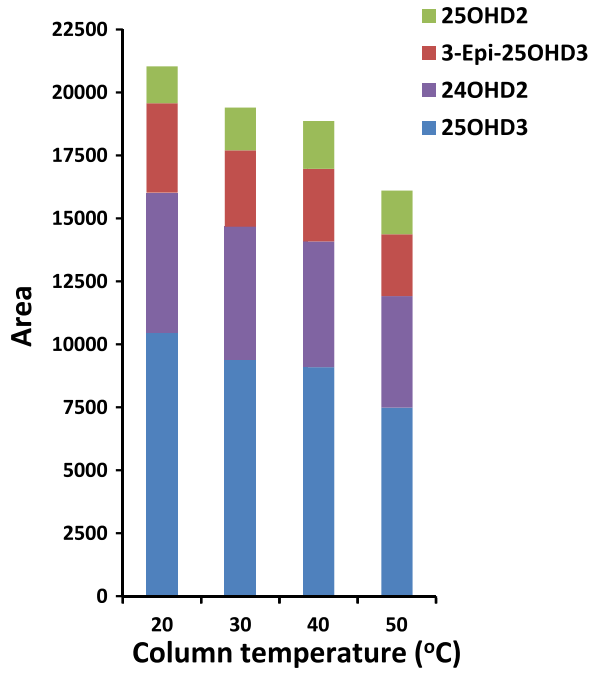


Fig. 11. Analyte areas of vitamin D metabolites with increasing column temperature (20–50 °C).

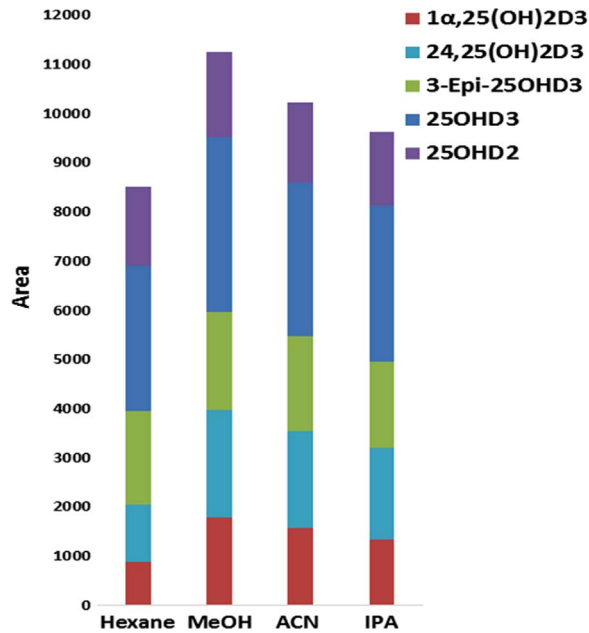


Fig. 12. Analyte areas with different solvents used for sample reconstitution.

## 2. Experimental design, materials, and methods

The sample preparation, UPSFC-MS/MS and UPLC-MS/MS methodologies for the data presented here have been previously described and cited [1]. The DMEQ-TAD sample preparation method is described previously [2].

## Acknowledgments

We would like to thank Prof. Cedric Shackleton for advice in developing methods. The study was supported by funding from a National Institute of Arthritis and Musculoskeletal and Skin Diseases award (R01 AR063910 to MH), a Royal Society Wolfson Merit Award (WM130118 to MH) and an Academy of Medical Science UK Newton Advanced Fellowship (NAF004\1002 to K.-H.S.).

## Transparency document. Supplementary material

Transparency document associated with this article can be found in the online version at <https://doi.org/10.1016/j.dib.2018.08.027>.

## References

- [1] C. Jenkinson, A. Taylor, K.H. Storbeck, M. Hewison, Analysis of multiple vitamin D metabolites by ultra performance supercritical fluid chromatography-tandem mass spectrometry (UPSFC-MS/MS), *J. Chromatogr. B* 1087–1088 (2018) 43–48.
- [2] M. Kaufmann, C. Gallagher, M. Peacock, K.P. Schlingmann, M. Konrad, H.F. DeLuca, R. Siqueiro, B. Lopez, A. Mourino, M. Maestro, R. St-Arnaud, J. Finkelstein, D.P. Cooper, G. Jones, Clinical utility of simultaneous quantitation of 25-hydroxyvitamin D & 24,25-dihydroxyvitamin D by LCMS/MS involving derivatization with DMEQ-TAD, *J. Clin. Endocrinol. Metab.* 99 (7) (2014) 2567–2574.